Computed Microtomography at GSECARS, 13-BM

Mark Rivers

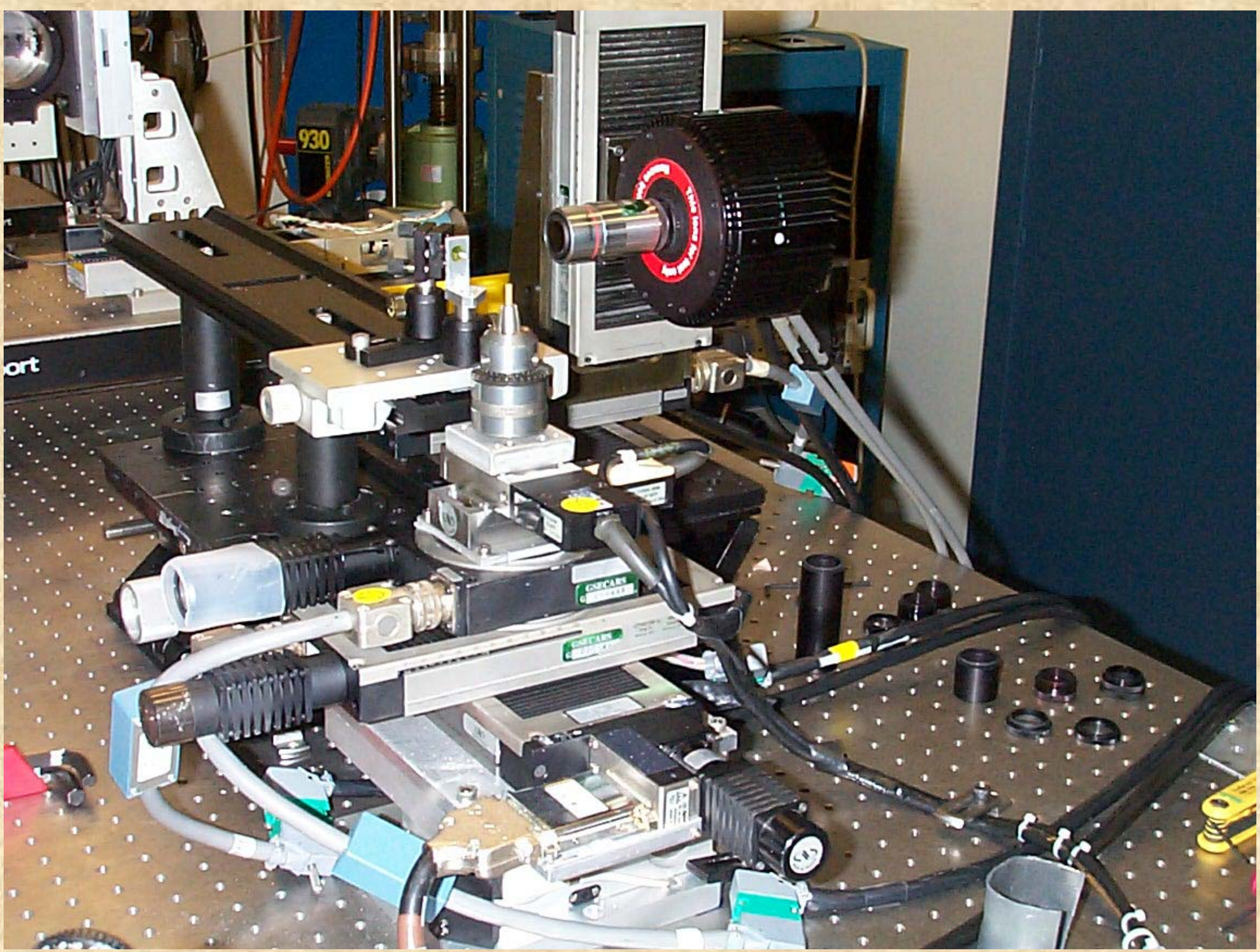
CARS

The University of Chicago

13-BM Absorption Tomography Setup

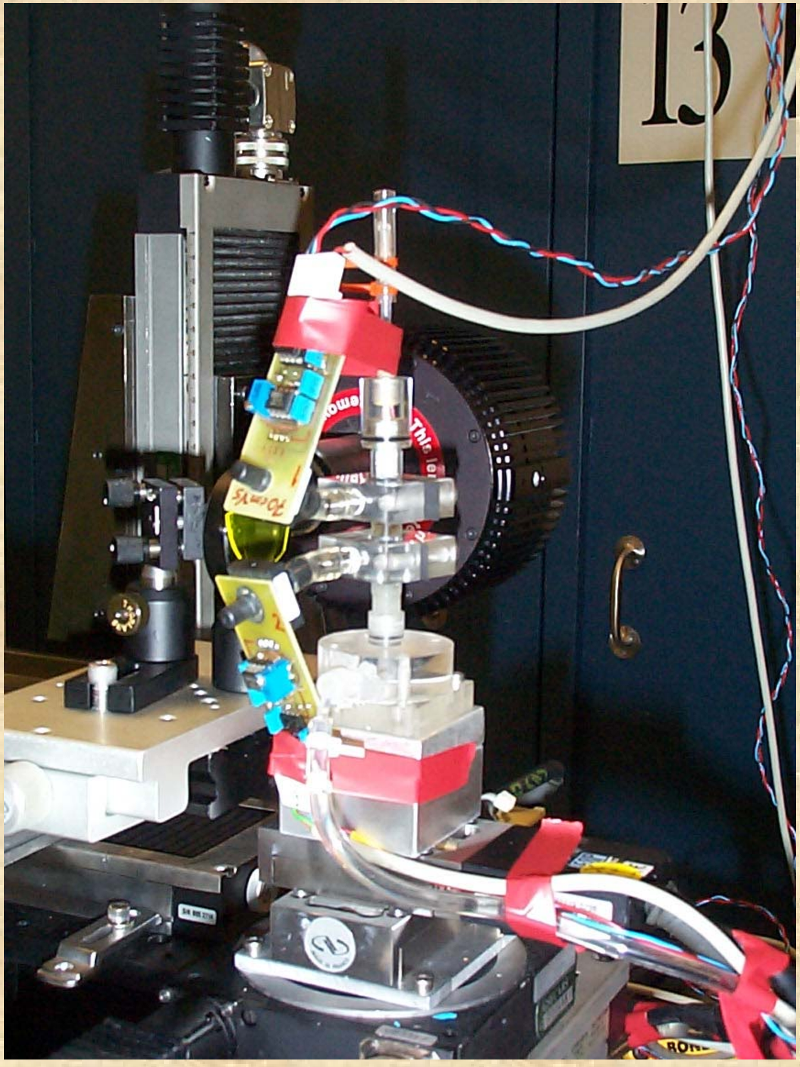
- Silicon 111 monochromator
  - 8-65 keV
  - 14-25mm fixed offset
  - Intensity feedback
- Vertical focusing mirror in 5:1 position, could be used for pink beam
- Roper CCD cameras
  - MicroMAX 5MHz, interline transfer, 7 micron pixels, 12-bits (2)
  - Pentamax 5MHz, mechanical shutter, 7 micron pixels, 12-bits (1)
  - ST-138, 1 MHz, mechanical shutter, 25 micron pixels, 16-bits (1)
- Optics
  - YAG single crystal scintillator (100-500 microns thick)
  - 5X to 20X Mitutoyu microscope objectives; zoom lens
  - Modular tubes to optimize resolution/field of view

Experiment Setup (13-BM-D)



Experiment Setup (13-BM-D)

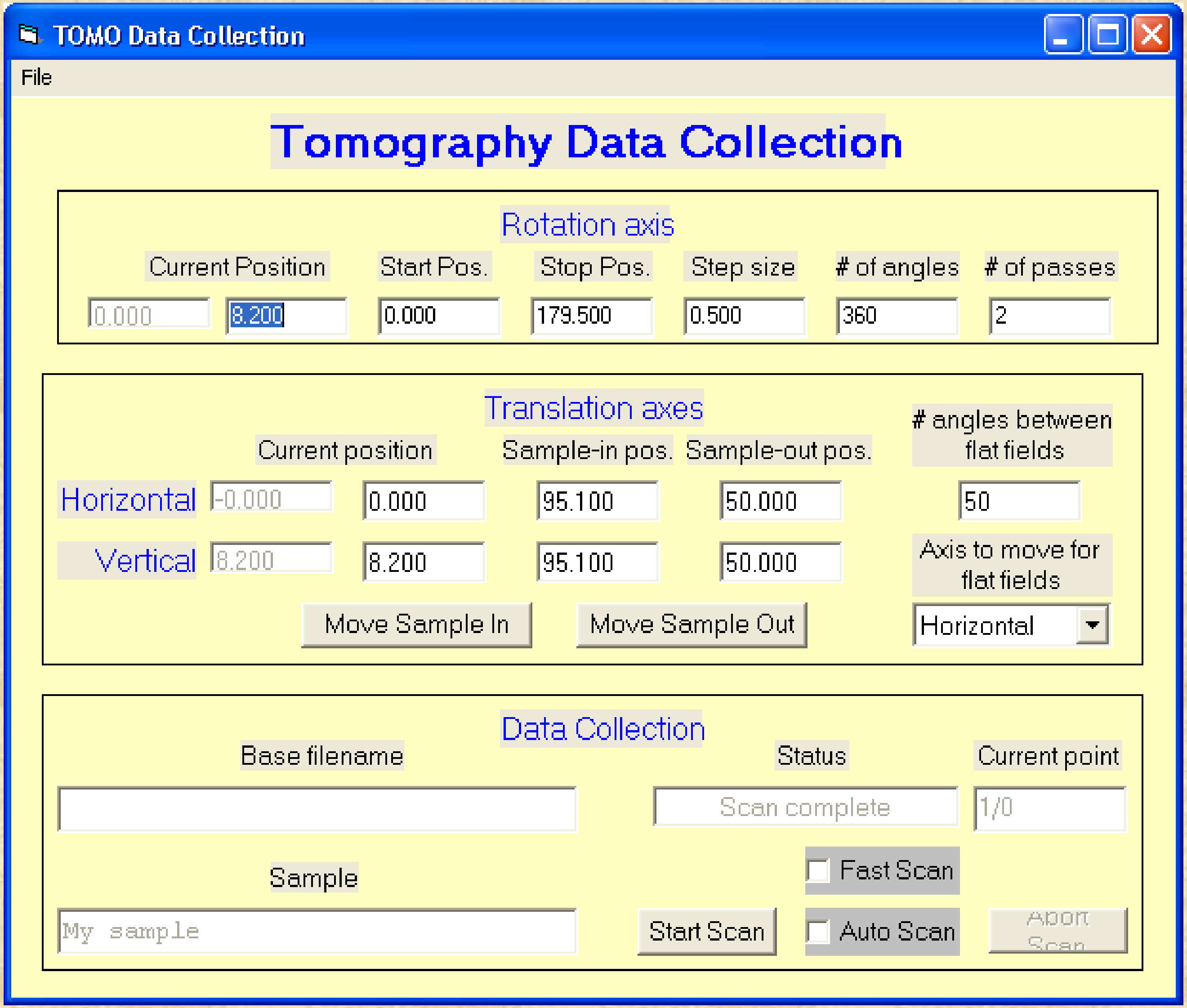
Soil Drainage Experiments (Dorthe Wildenschild, Oregon State)



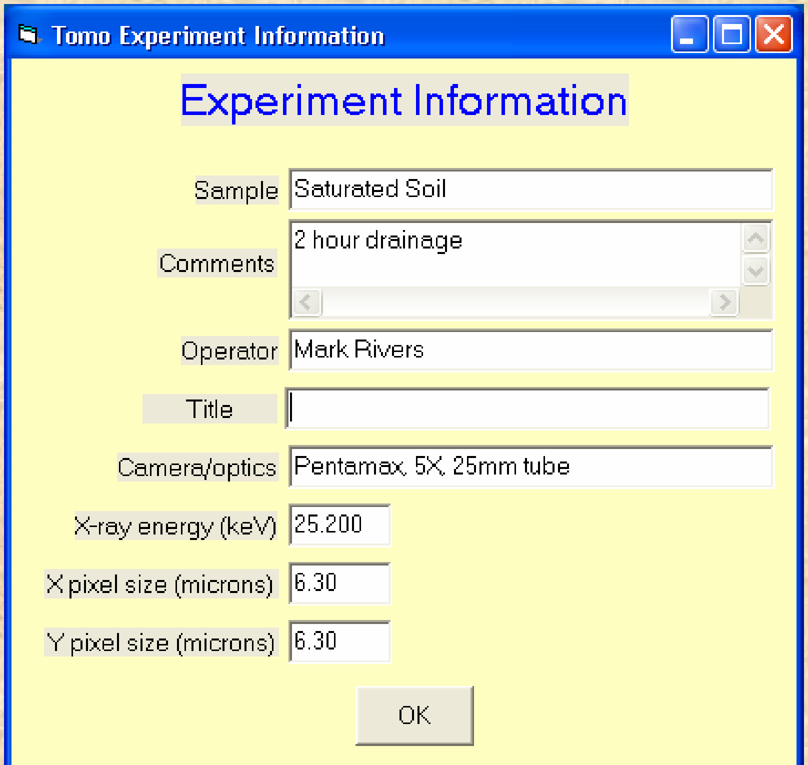
Data Collection

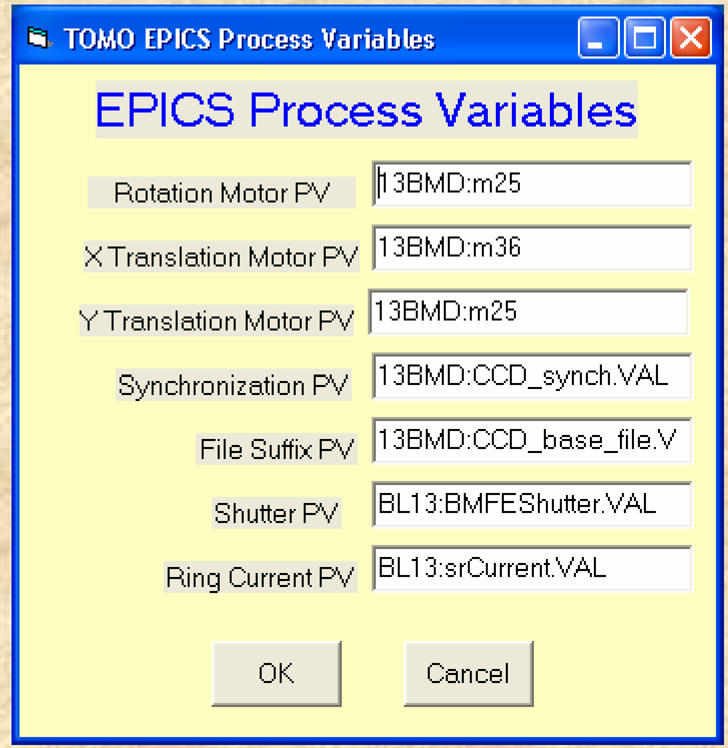
- Visual Basic program under Windows is the data collection GUI
    - May migrate to Python in the future
- Communicates with:
    - Roper Scientific WinView program to collect and save frames via COM (aka OLE) interface
    - EPICS to move motors, read beamline status
- Program is a “server” that knows how to collect a single tomography data set

Data Collection Program



Data Collection Program

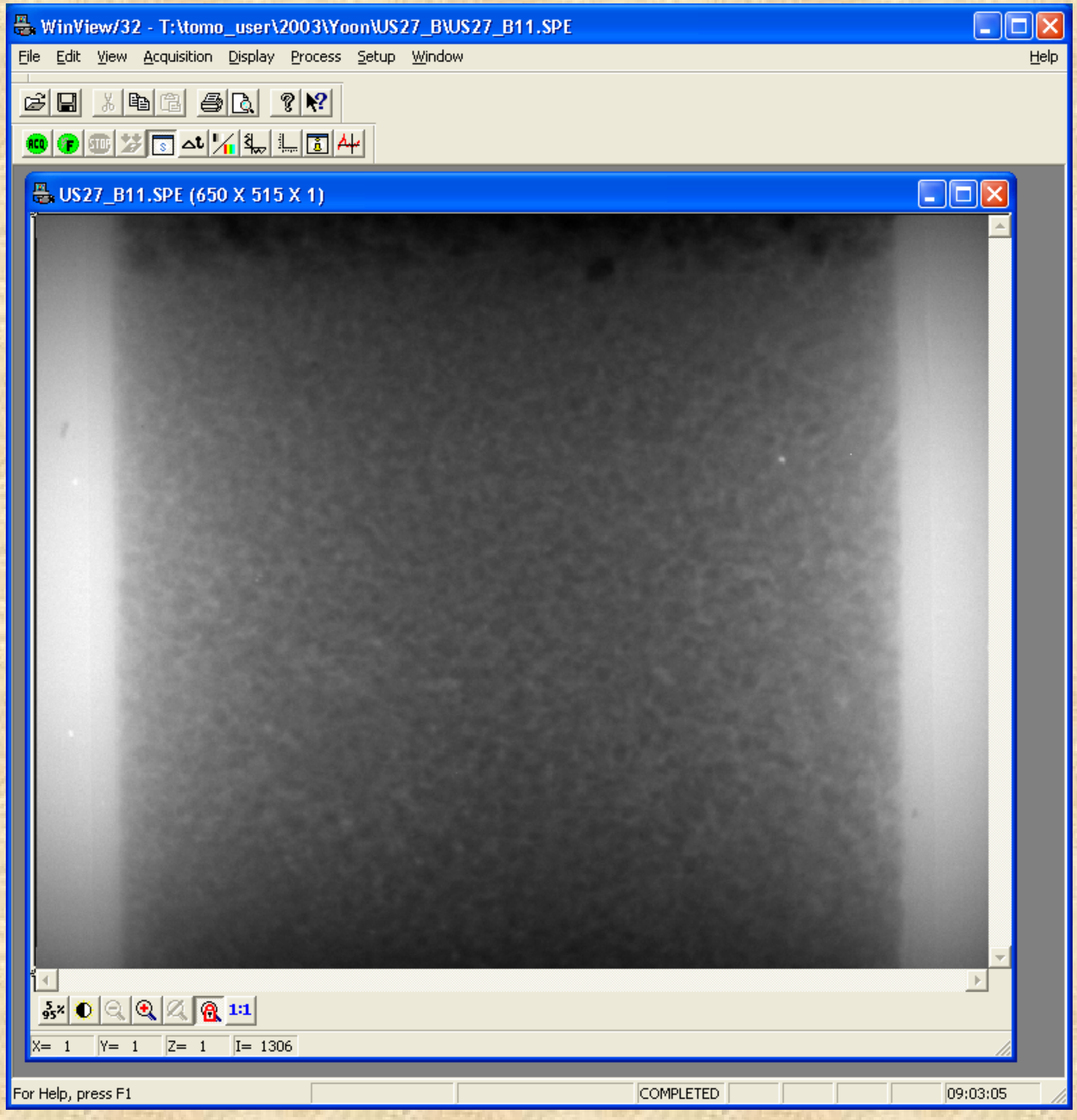




WinView

Primary user interface for focusing, setting collection time, etc.

Collects and saves frames under control of Visual Basic application



Data Collection

- “Clients” (typically IDL) can perform other operations (change energy, change vertical sample position, etc.) and then tell the “server” to collect a new data set.
    - Communication of file name and start command through EPICS
- Most users want to image a lot of samples, use 2x2 binning for 650x510 pixel frames, 720 frames (360 if speed is more important)
- Exposure times typically 0.5-5 seconds
- Collection overhead of ~2 seconds if collect single .SPE files, 15-30 minutes per data set
- Can reduce overhead by having WinView collect all frames in a single file, 7 minutes for full 720 angle data set.
- Would like to get under 1 minute for some applications

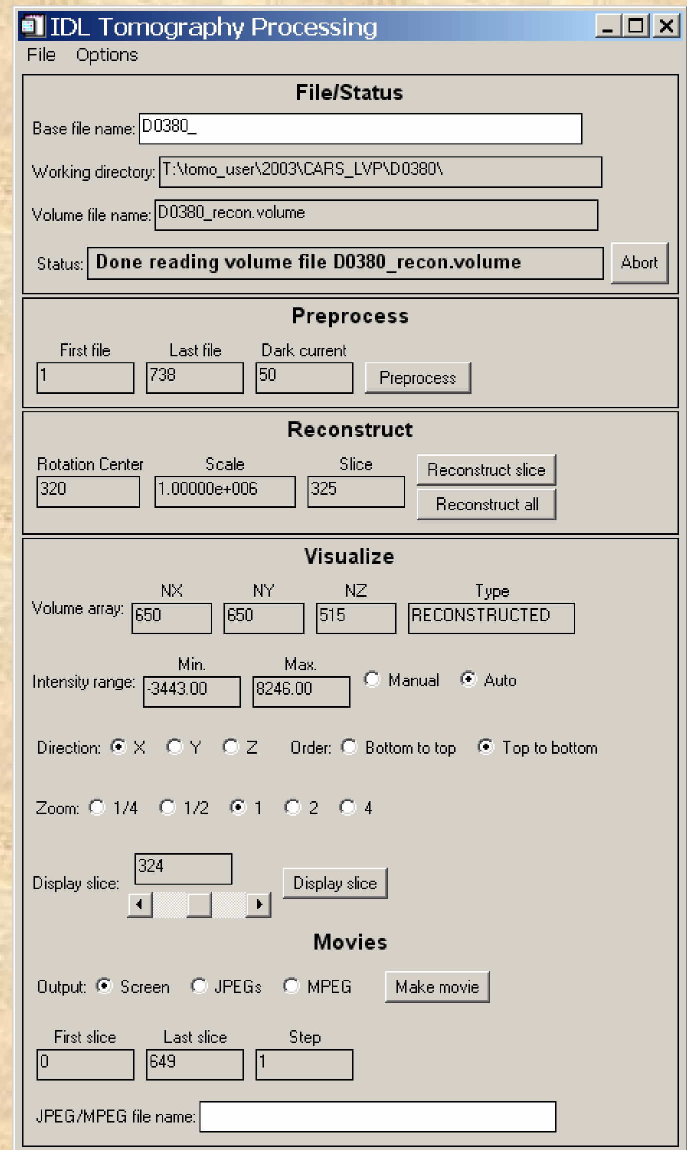
Data Collection

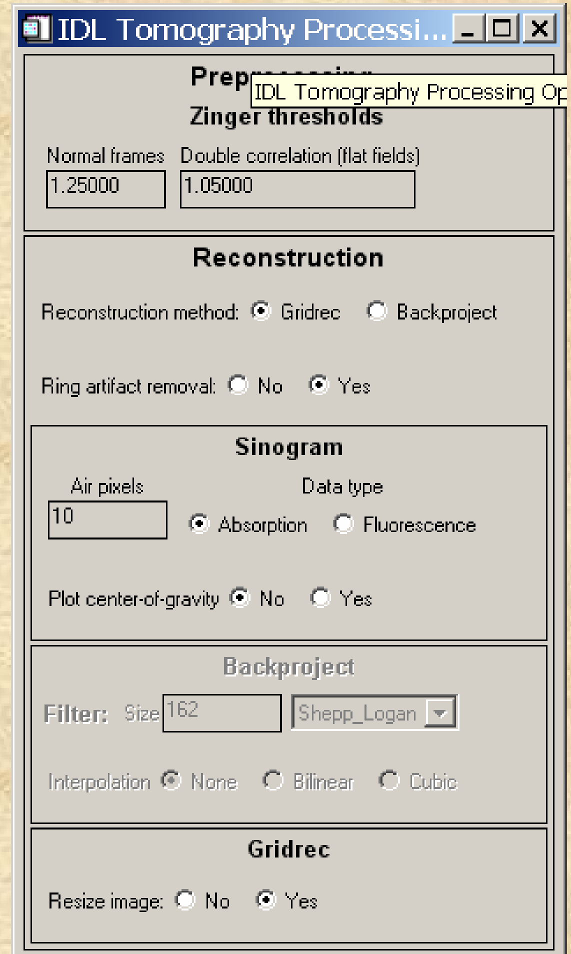
- 1 Terabyte RAID array on Windows 2000 server
- Super-DLT tape robot with 7 on-line tapes for backup and archiving
- DVD writers for user-data
- 1000 Base-T Ethernet just installed between collection machine, server and analysis machines
- Data format
  - Multiple .SPE files or single large .SPE file for raw data
  - Preprocessing creates a 3-D (X-Y-Theta) file in netCDF format
  - Reconstruction creates a 3-D (X-Y-Z) file in netCDF format
  - All files are 16-bit integers

Data Processing

- IDL GUI
- Preprocessing done in IDL
  - Dark field, flat field, zinger removal, ring artifact reduction
  - Sinogram creation
- Calls C code for reconstruction
  - "Gridrec" FFT algorithm from BNL
  - Enhanced to use Intel Math Kernel Library on Windows and Linux
  - Both IDL code and MKL FFT routines are multi-threaded
- Reconstruction times on 3GHz Pentium 4:
  - 650 pixels x720 angles: 0.5 sec/slice, 4 minutes/volume
  - 1300 pixels x 1440 angles: 1.9 sec/slice, 35 minutes/volume
- Have 4-processor 2GHz Linux box that can also be used

Data Processing

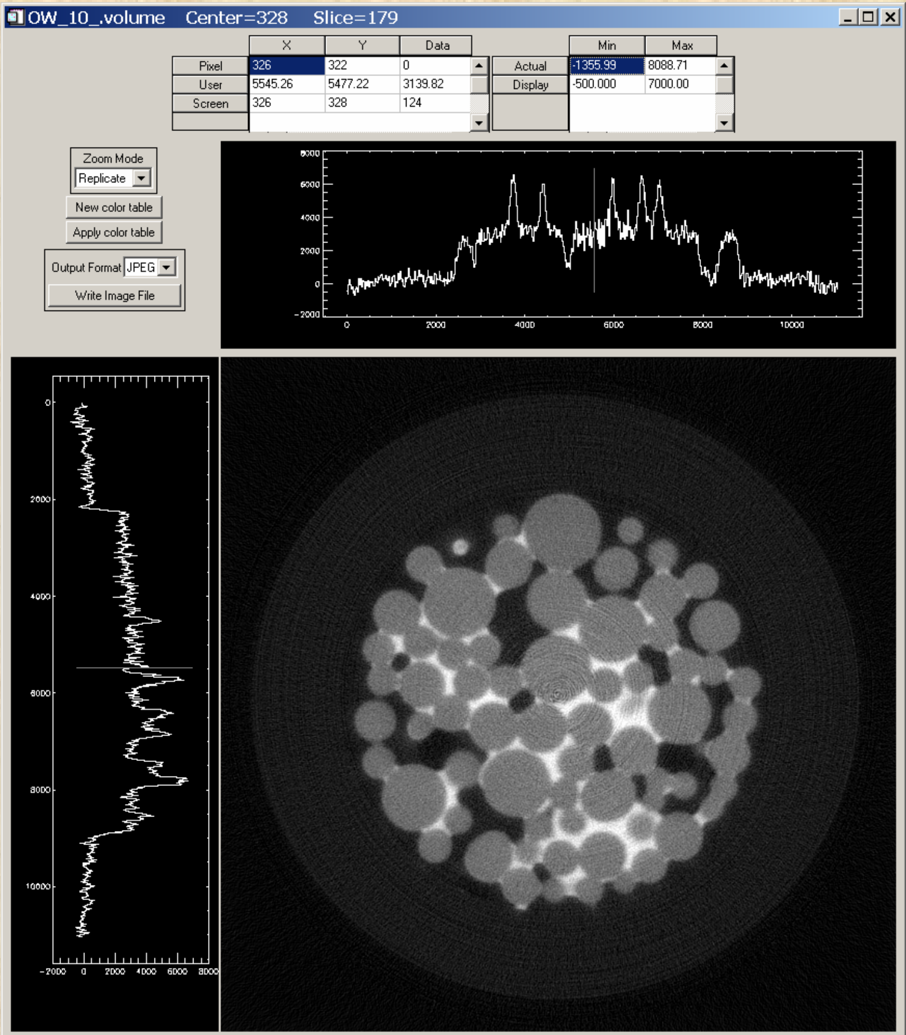




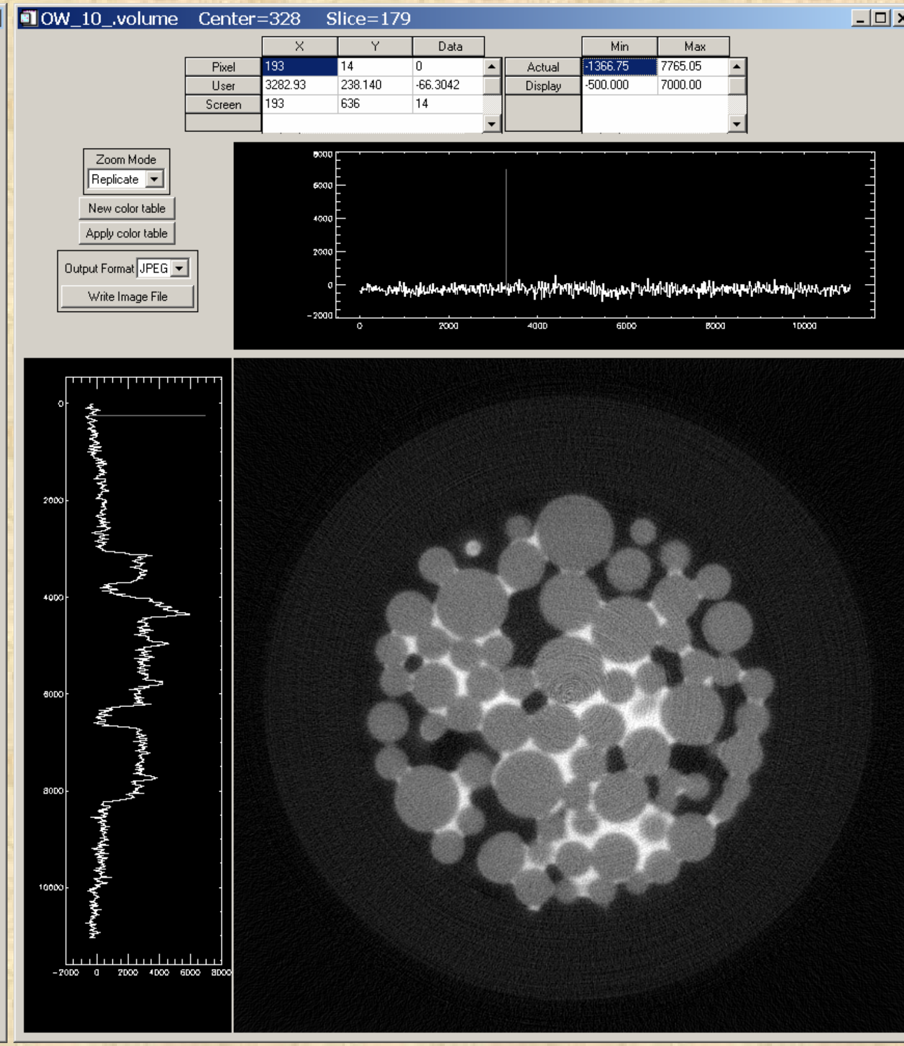
Ring Artifacts

- Compute “average” row of sinogram (i.e. sum down columns)
- This should be very smooth
- High-frequency content is assumed to be pixel anomalies
- Low-pass filter the average row
- Subtract average row from low-pass filtered version
- The differences are pixel anomalies that are then added back in to the data in each row of the sinogram
- Low-pass filter width is adjustable

Ring Artifacts



Without Reduction



With Reduction

Quantitative Analysis Needs

- Good segmentation tools that are “as good as your eye”
- Basic statistics
- Brent Lindquists 3DMA can be used for some metrics
- Conference at LSU this September to bring together tomography data collection folks with mathematicians

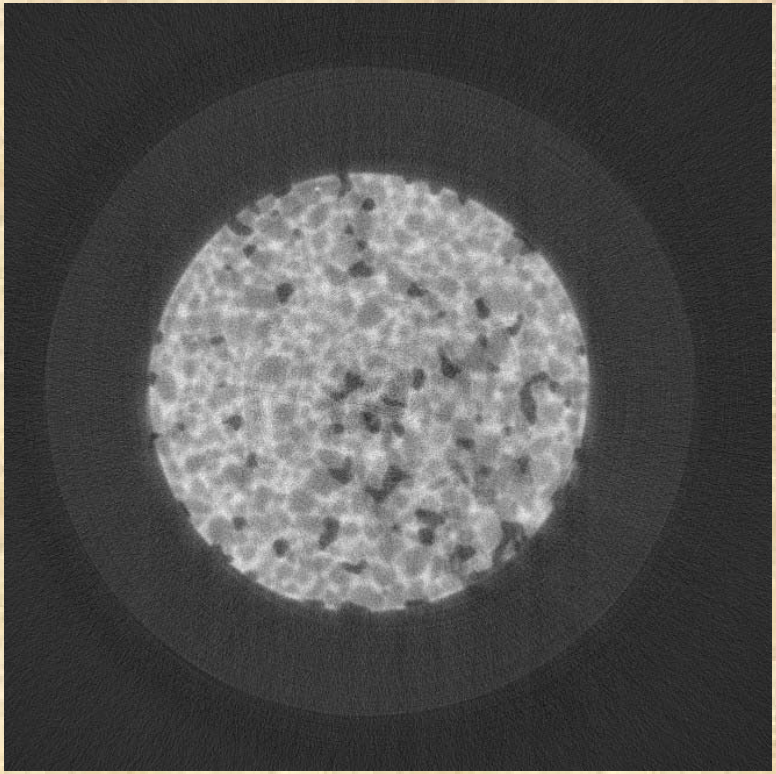
Absorption Tomography Examples

- Glass/epoxy chips, 6mm diameter. Dogan Paktunc, CANMET: radiograph movie and reconstruction movie
- Eocene snail fossil, 20 mm tall. Becca Price, University of Chicago: movie
- Pumice sample. Fred Anderson, University of Chicago. Movie
- Garnet porphyroblast development in metamorphic rocks. Richard Speiss, University of Padova. Movie.
- Meteorites (chondrules, calcium-aluminium inclusions). Movie.
- Soil aggregates. Iian Young, Scotland. Movie.
- Nickel battery mesh. Jim Brown, CANMET. Movie

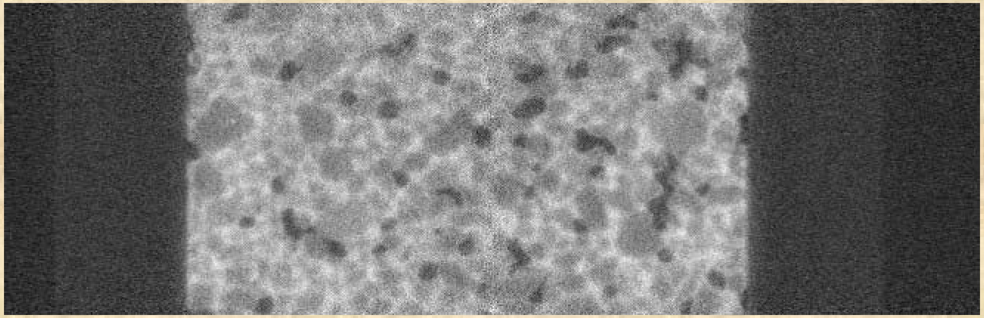
Tomography of soil drainage

6mm diameter soil cores, saturated with KI solution, then drained

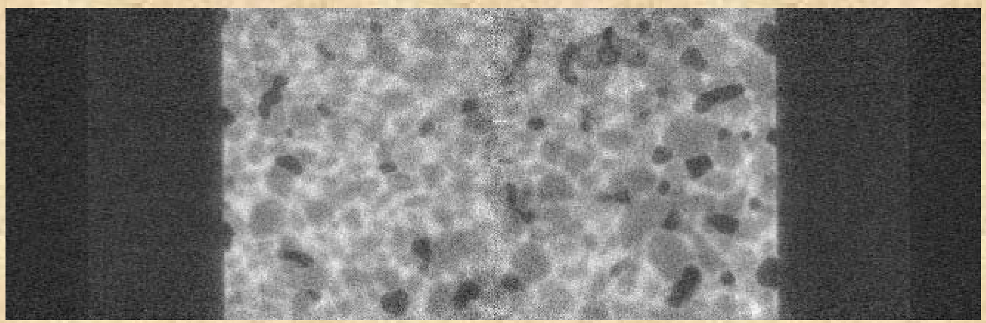
Imaged just above I K-edge at 33 keV



Z slice



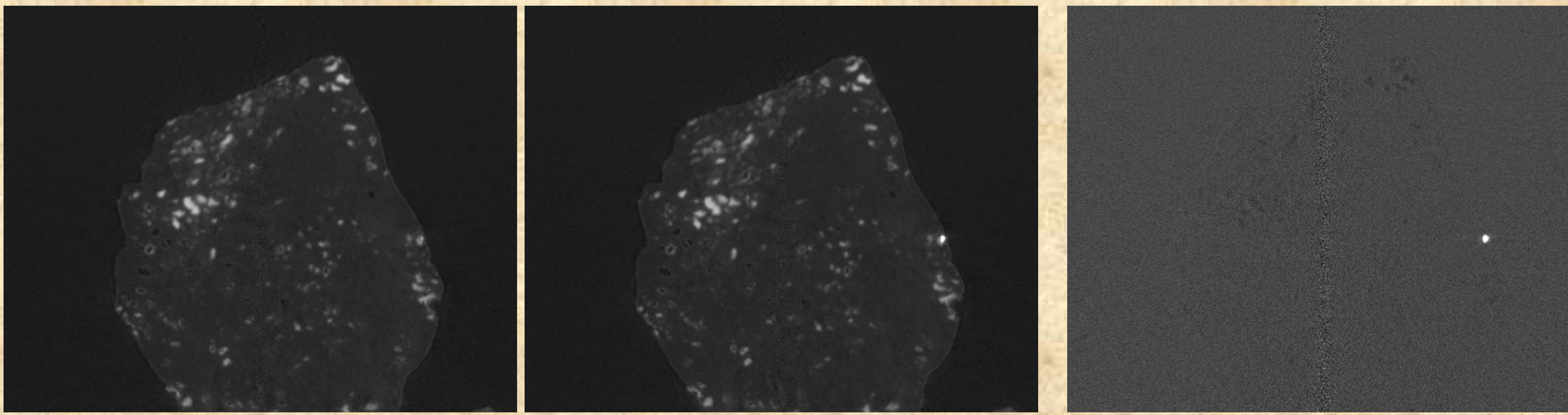
X slice



Y slice

Carbonado diamonds – looking for zircons above and below Zr K edge

Zircon is used for dating – need to find crystals in a very hard, opaque matrix



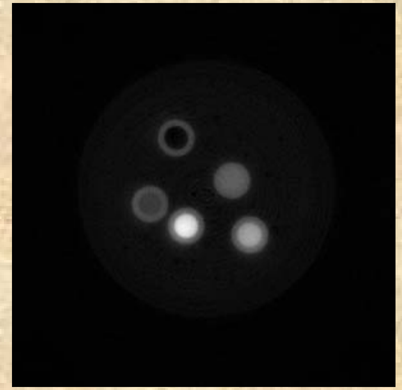
100 eV below Zr K absorption edge

100 eV above Zr K absorption edge

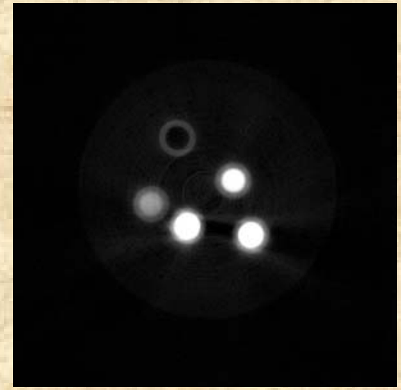
Difference of above and below edge images

Differential Absorption Tomography

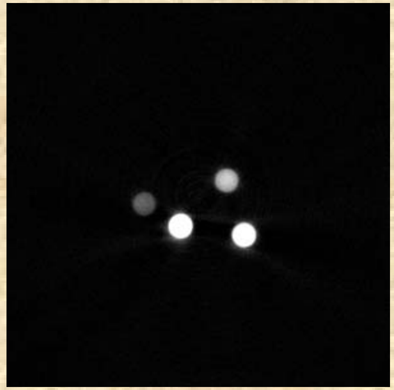
- Collect 2 absorption data sets, above and below the absorption edge of the element of interest – also fast
- Requires a substantial change in linear attenuation coefficient due to element of interest
  - Major elements, not trace elements
- Example: 2mm capillaries with KI solutions, varying concentration



33.1 keV, below I edge



33.2 keV, above I edge

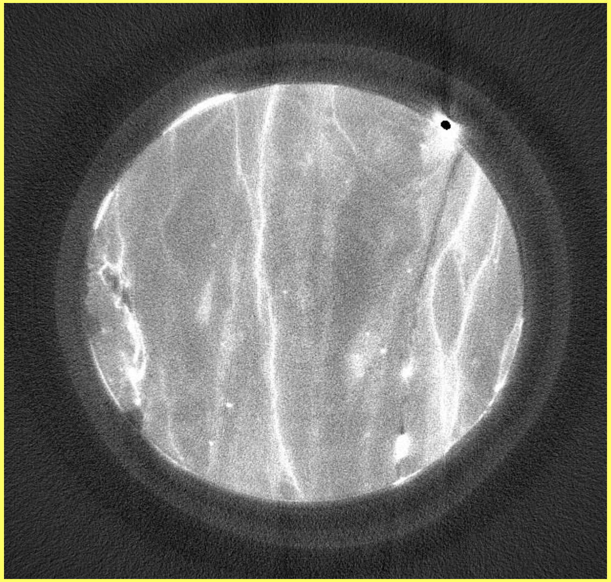


Difference

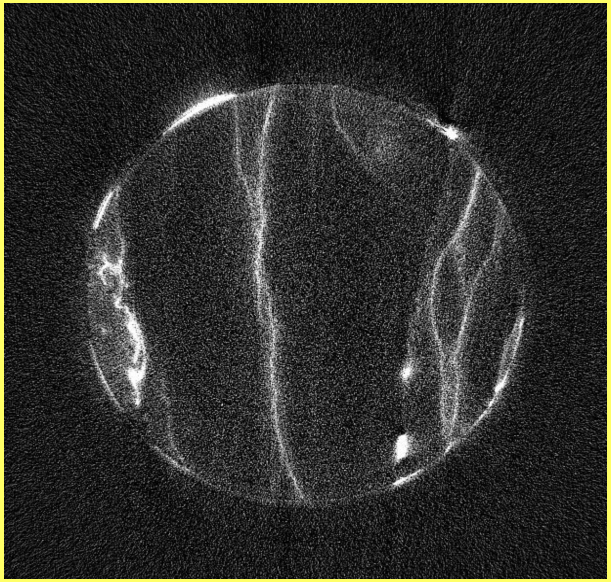
Differential Absorption Tomography

1cm diameter rock core was saturated with CsCl solution and images were taken above and below the Cs K absorption edge (36 keV). Waste repository rock (Susan Altman, Sandia)

36.0 keV, below Cs K absorption edge
(movie)



36.2 keV, above Cs K absorption edge
(movie)

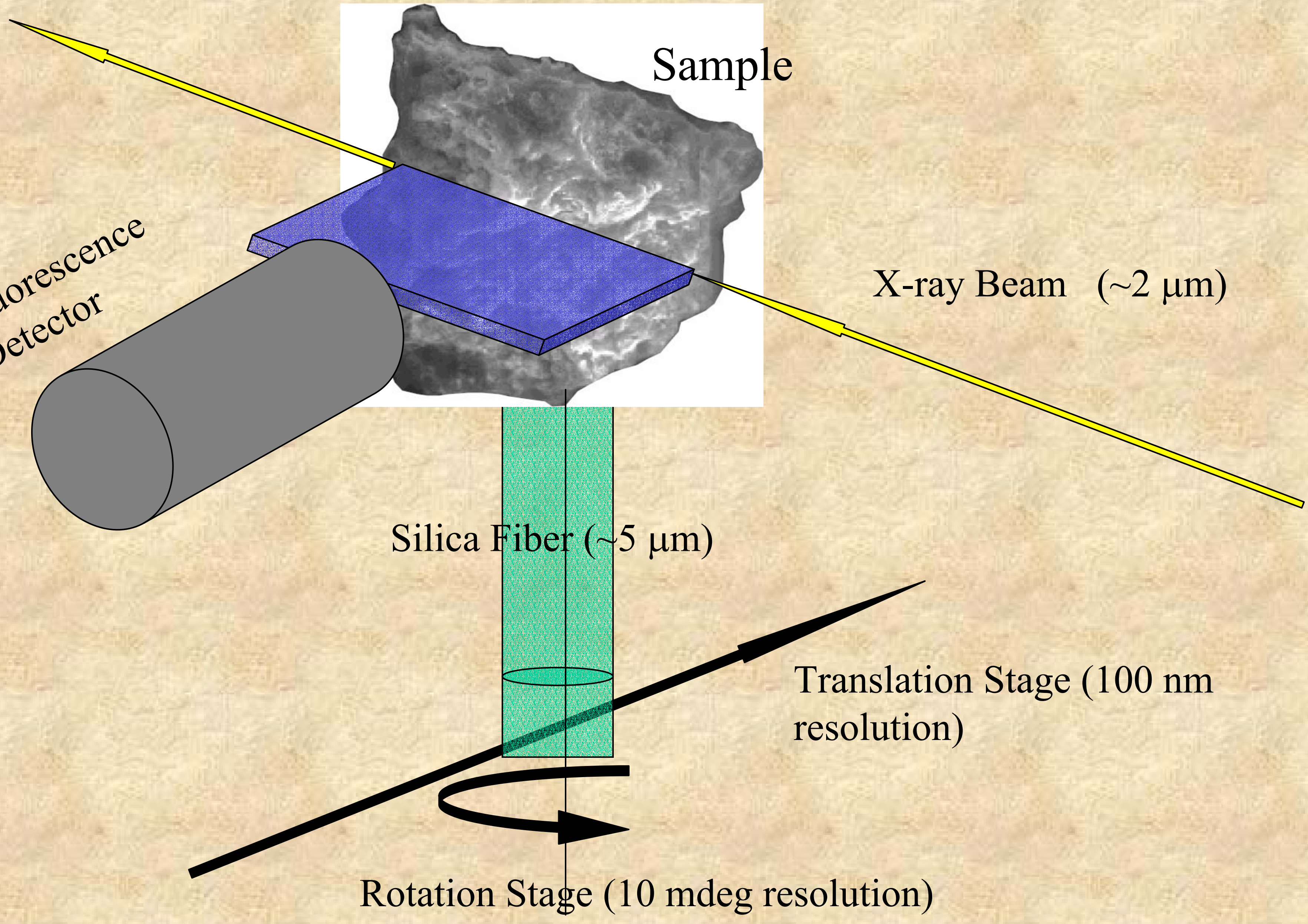


Difference of above and below edge images
(movie)

Fluorescence Microtomography

- Images of the internal distribution of specific elements
- Synchrotron XRF is very sensitive, sub-ppm
- Valuable when object cannot be sectioned at required resolution
- Absorption must not be too large
- First generation (pencil beam)
  - Requires NX translations and NR rotations for each slice.
  - Slow - similar to conventional 2-D map
- APS Undulator Source
  - Less than 1mm vertical by 3mm horizontal
  - 1000 times more intense monochromatic beam than bending magnet

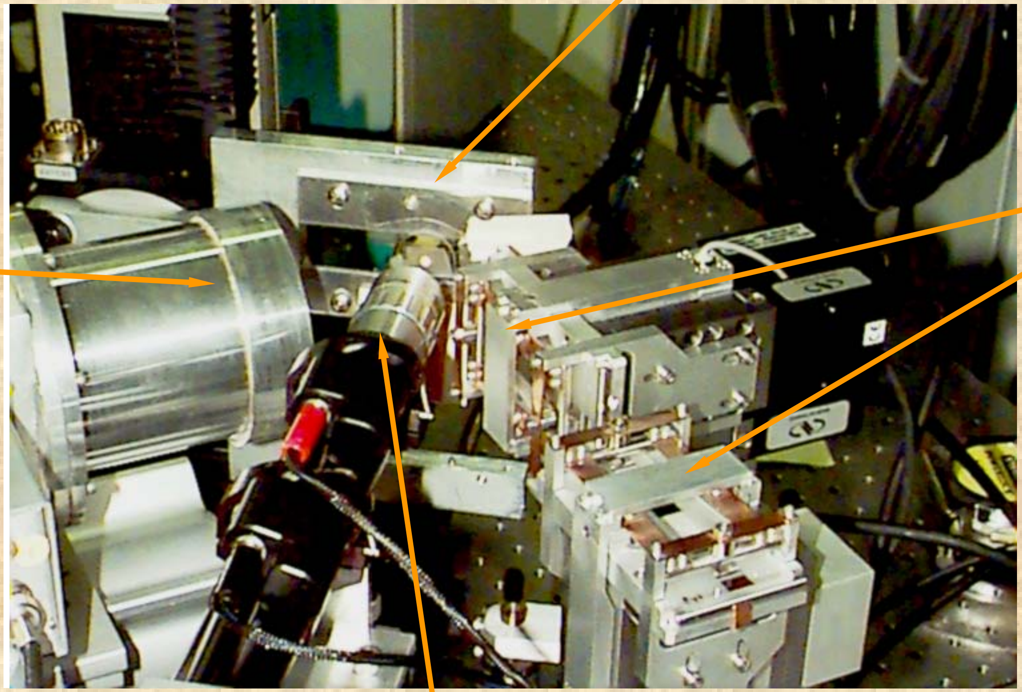
Fluorescence Microtomography Apparatus



The GSECARS Fluorescence Microprobe/Microtomography Apparatus

Sample x-y-z-θ stage: 0.1μm step sizes

Fluorescence detectors: Multi-element Ge detector Lytle Chamber, Si(Li) detector, or Wavelength Dispersive Spectrometer

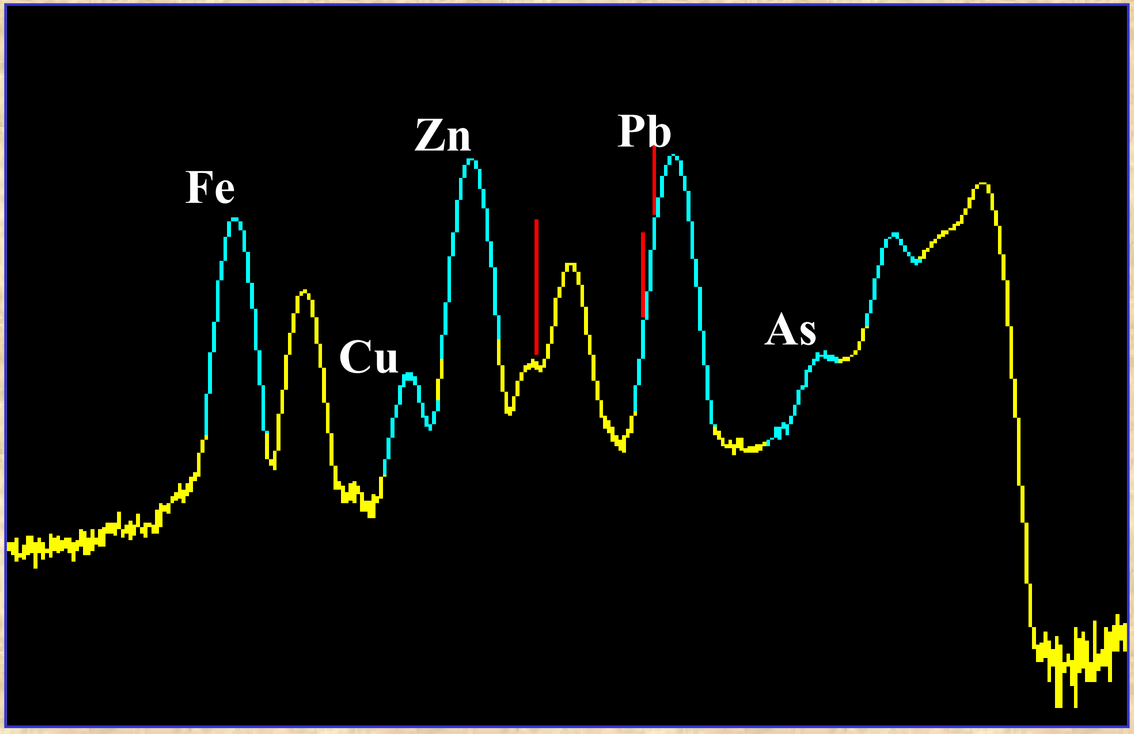


Horizontal and Vertical Kirkpatrick-Baez focusing mirrors

Optical microscope (10x to 50x) with video system

Data Collection Setup: Fluorescence Detector

- Regions of Interest (ROIs) are selected for each element of interest
- Fluorescence intensity in each peak is collected simultaneously at each pixel.

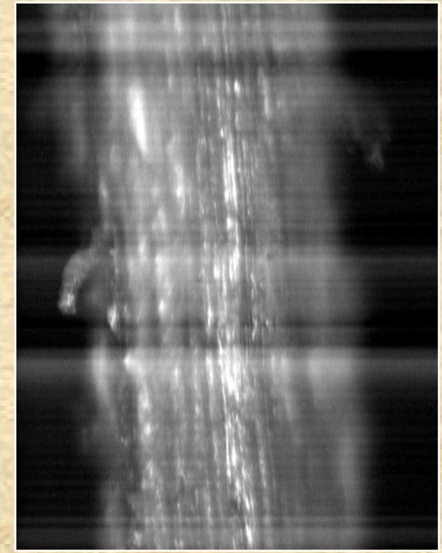


X-ray Fluorescence Spectrum

(logarithmic display of Detector 7)

Fluorescence Tomography: Sinograms

The Raw fluorescence tomography data consists of elemental fluorescence (uncorrected for self-absorption) as a function of position and angle: a ***sinogram***. This data is reconstructed as a virtual ***slice*** through the sample by a coordinate transformation of (x,ω) -> (x, y). The process can be repeated at different z positions to give three-dimensional information.



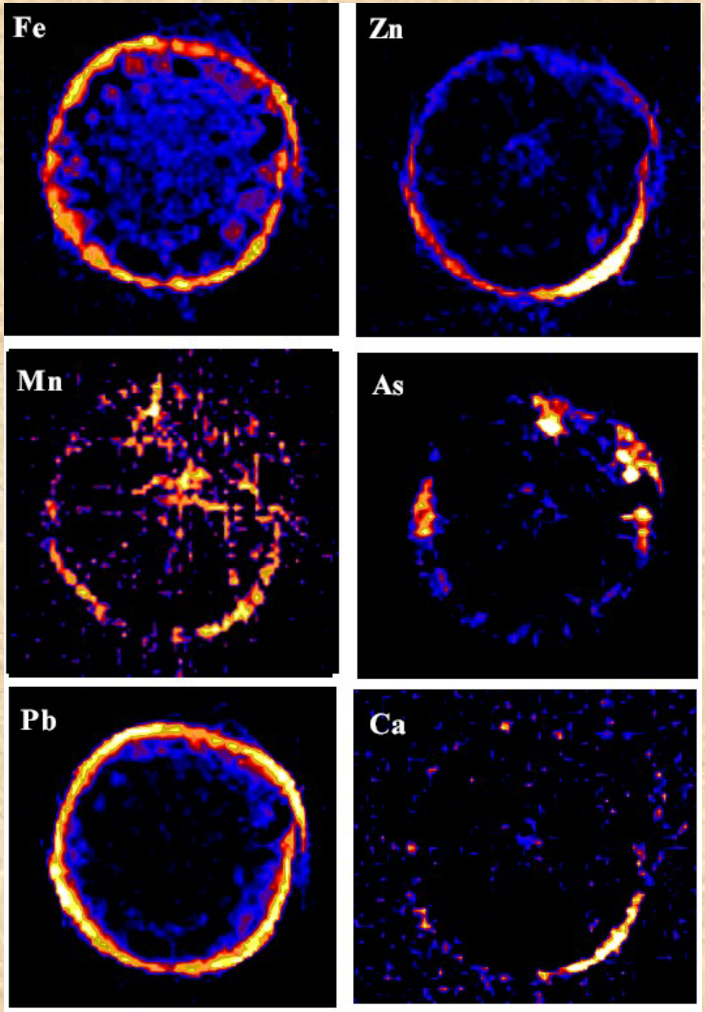
Fluorescence Sinograms for Zn, Fe, and As collected simultaneously for a section of contaminated root (photo, right):

x: 300μm in 5μm steps ω: 180° in 3° steps

Fluorescence Tomography: Distributions of Heavy Metals in Roots

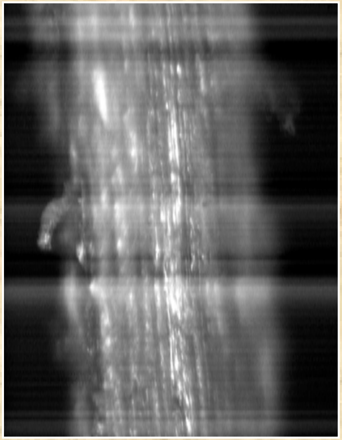
S. Fendorf, C. Hansel (Stanford): **Toxic Metal Attenuation by Root-borne Carbonate Nodules**

CT images of a 300 micron root cross section (*Phalaris arundinacea)* shows Fe and Pb uniformly distributed in the root epidermis whereas Zn and Mn are correlated with nodules. Arsenic is highly heterogeneous and poorly correlated with the epidermis suggesting a non-precipitation incorporation mechanism.



Such information about the distribution of elements in the interior of roots is nearly impossible to get from x-y mapping alone:

Physically slicing the root causes enough damage that elemental maps would be compromised.

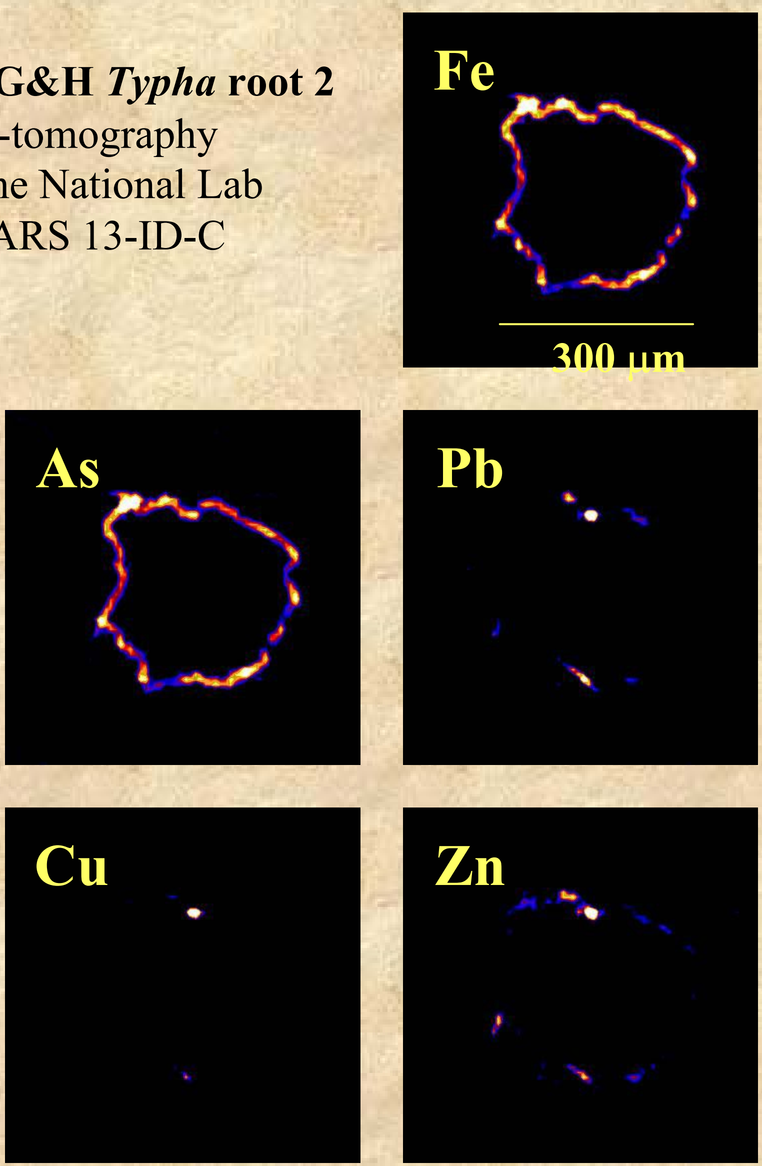


Photograph of root section and reconstructed slices of fluorescent x-ray CT for selected elements.

Fluorescence Tomography: Distribution of Arsenic in Cattail Roots

Nicole Keon, Harold Hemond (MIT):

Wells G&H *Typha* root 2
XRF μ-tomography
Argonne National Lab
GSECARS 13-ID-C



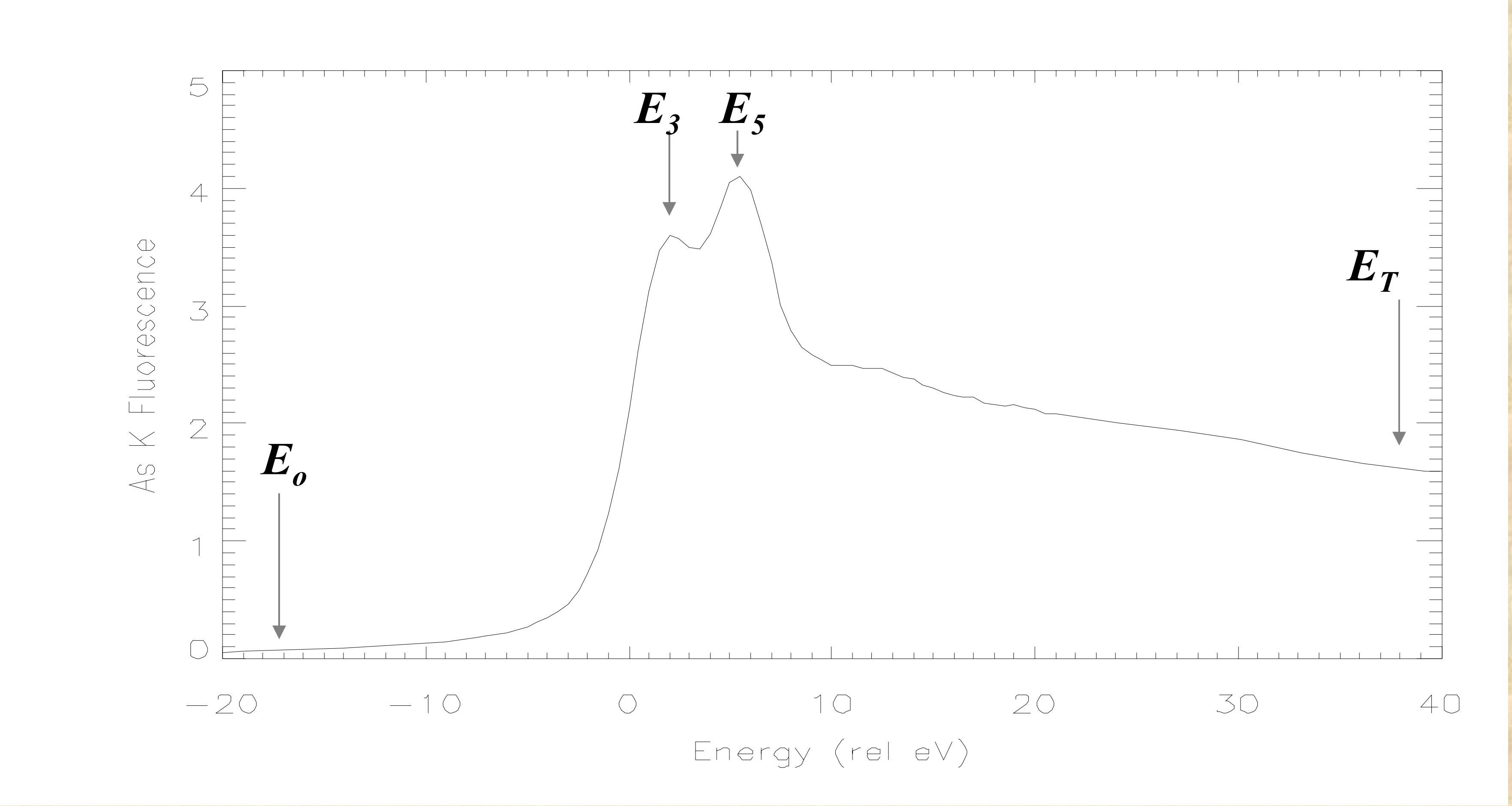
Studying a Superfund site (Wells G+H wetland) that gained notoriety in *A Civil Action*, a reservoir of approximately 10 tons of arsenic within the upper 50 cm of the sediment profile.

Most of the arsenic is sequestered in the wetland peat sediments with relatively little in the groundwater.

In contrast riverbed sediments in the wetland (5 feet away) have higher concentrations of aqueous (mobile) arsenic despite lower solid phase concentrations.

Hypothesize that the metabolic activity of the wetland plants may help to explain the sequestration of arsenic in the wetland.

Choice of Energies for Oxidation State Tomography (As^{3+}, As^{5+})

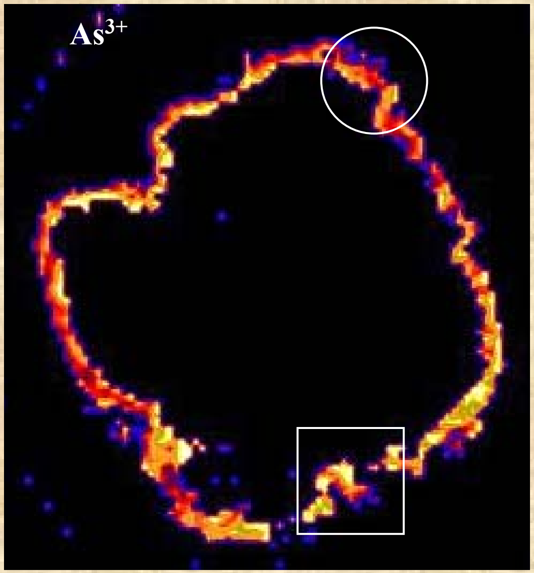


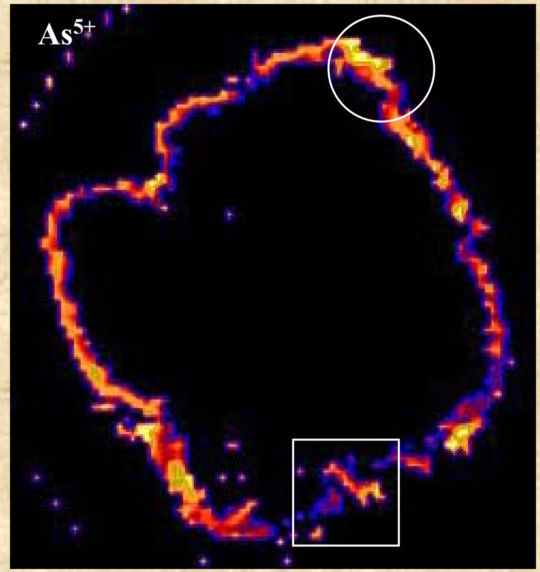
Fluorescence Tomography: Distribution of Arsenic in Cattail Roots

Nicole Keon, Harold Hemond (MIT):

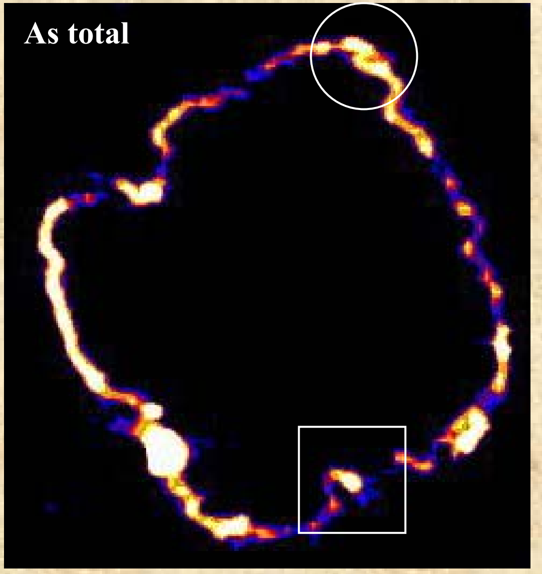
Typho_wet_oil_tomo7.001 (As^{3+} **energy) and .002 (total As energy).**

The As^{3+} image was produced by taking the ratio of the .001 and .002 images and assuming that ratio is 0.3 for 0% As^{3+} and 2.5 for 100% As^{3+}. The As^{5+} image was obtained as (1 - As^{3+} image).





Weighted redox means: As^{3+}=43%; As^{5+}=57%. As^{3+}/As^{5+} is generally heterogeneous (e.g., boxed area) and there is a tendency for As^{5+} to be on the exterior (e.g., circled area).

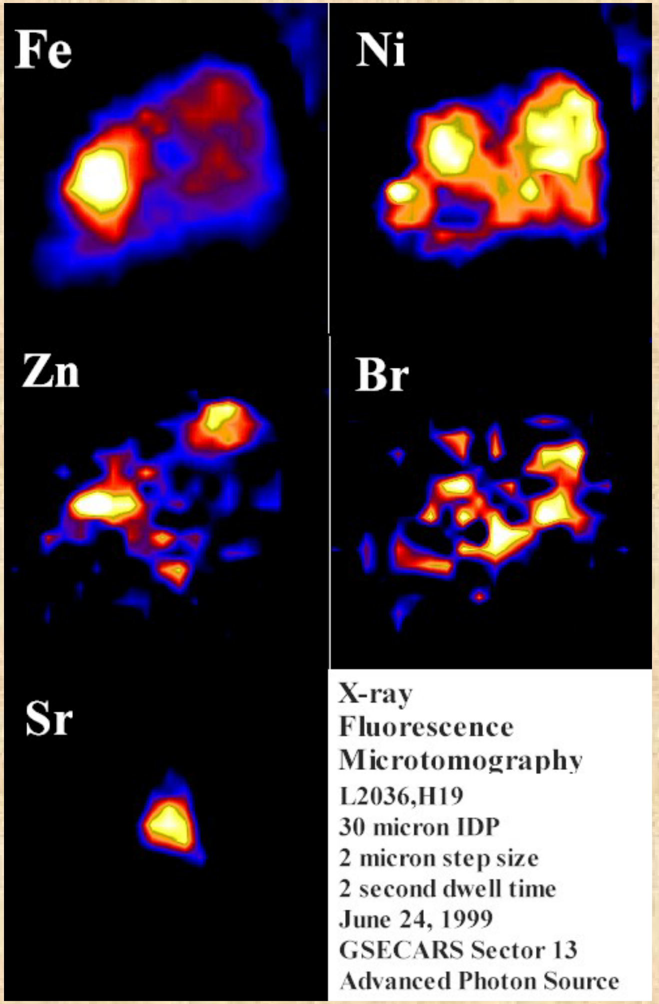


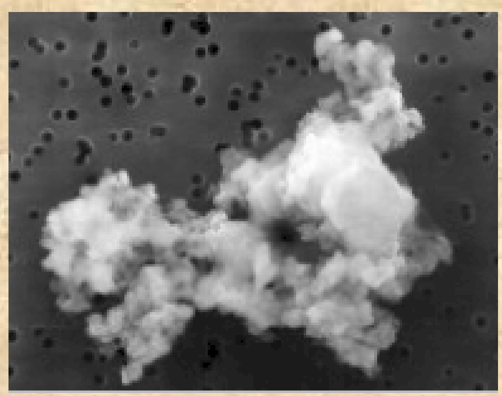
Fluorescence Tomography: Interplanetary Dust Particles

G. J. Flynn (SUNY, Plattsburgh): **Volatile elements in interplanetary dust**

Interplanetary Dust Particles (IDPs) collected by NASA aircraft from the Earth's stratosphere allow laboratory analysis of asteroidal and cometary dust.

MicroXRF analyses show enrichment of volatile elements, suggesting the particles derive from parent bodies more primitive than carbonaceous chondrites (Flynn and Sutton, 1995). The IDP fluorescence tomography images show that volatile elements (Zn and Br) are not strongly surface-correlated, suggesting that these elements are primarily indigenous rather than from atmospheric contamination



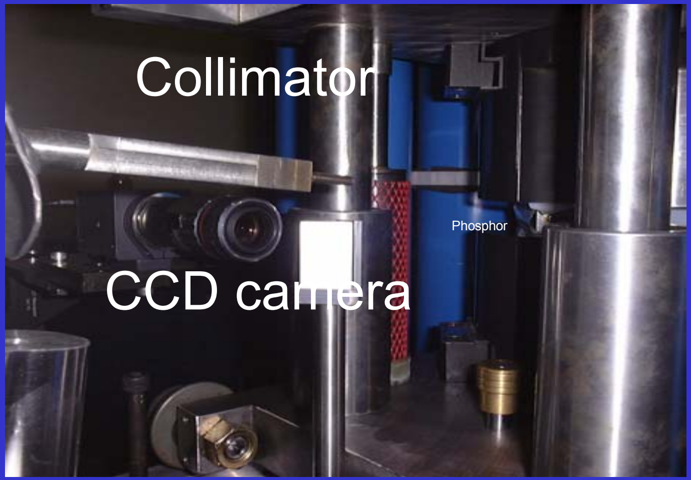


Towards High Pressure Tomography

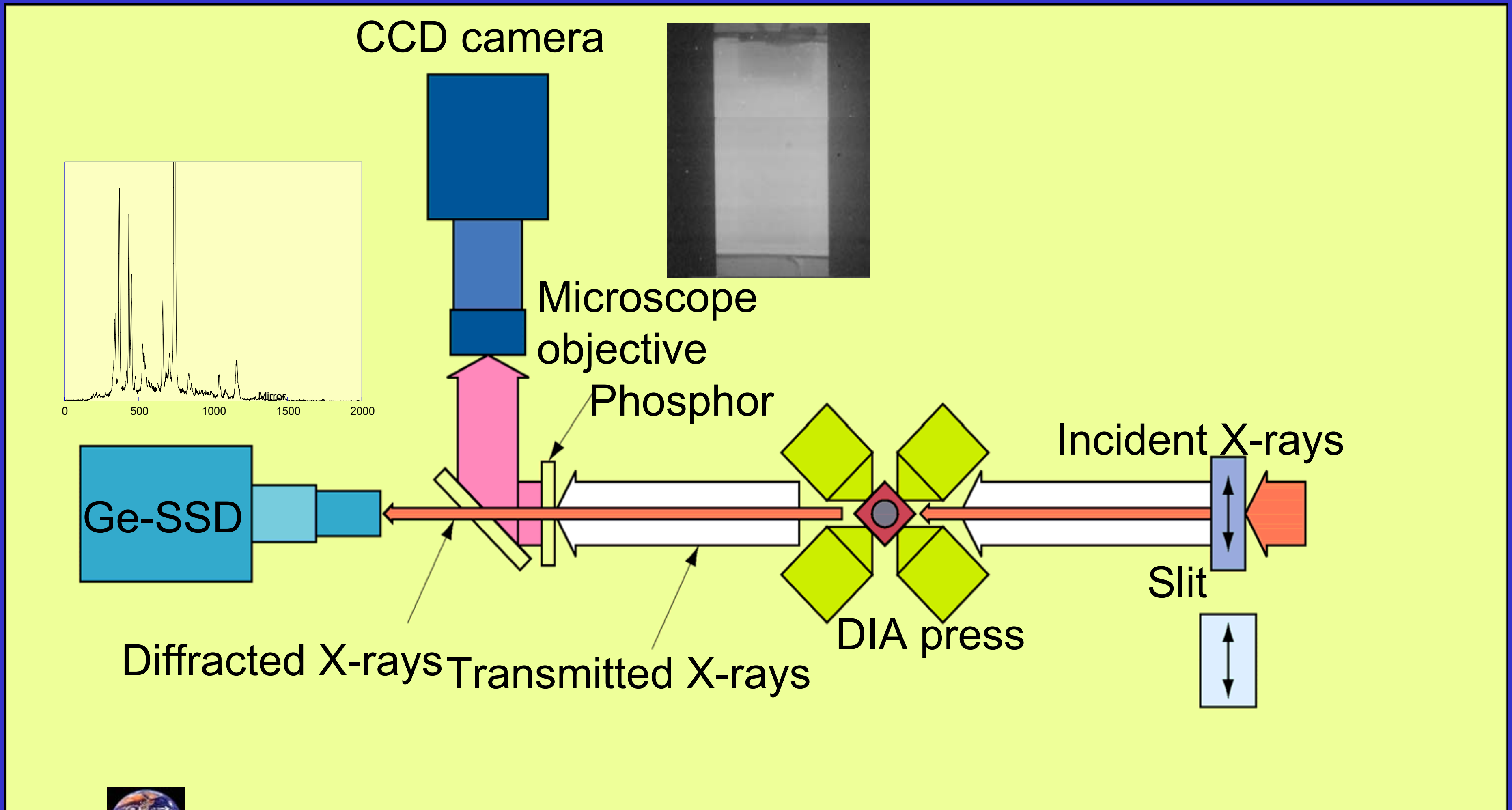
Yanbin Wang
Takeyuki Uchida
Mark L. Rivers
Stephen R. Sutton

GSECARS, Univ. Chicago

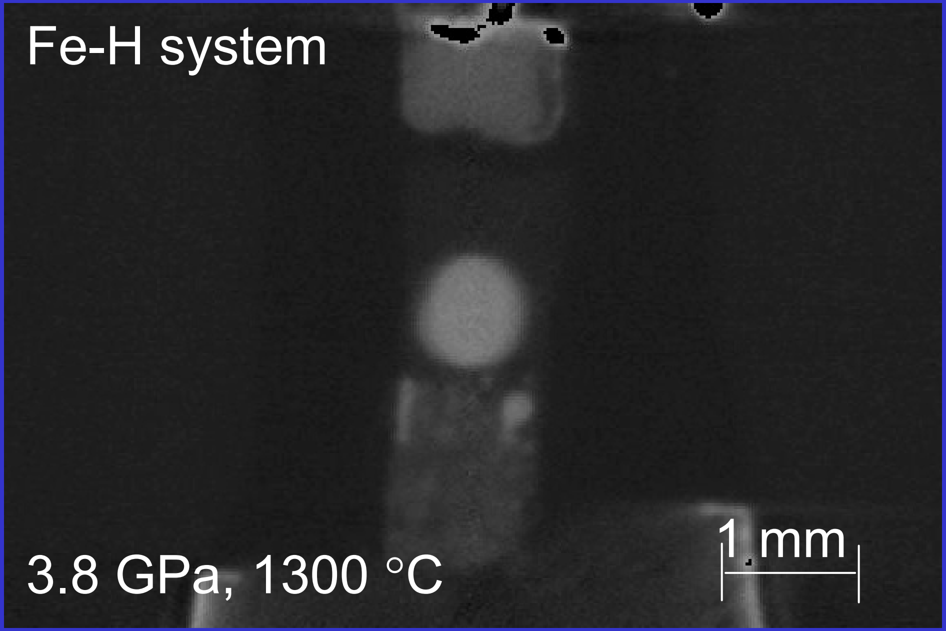
High Pressure Experimentation

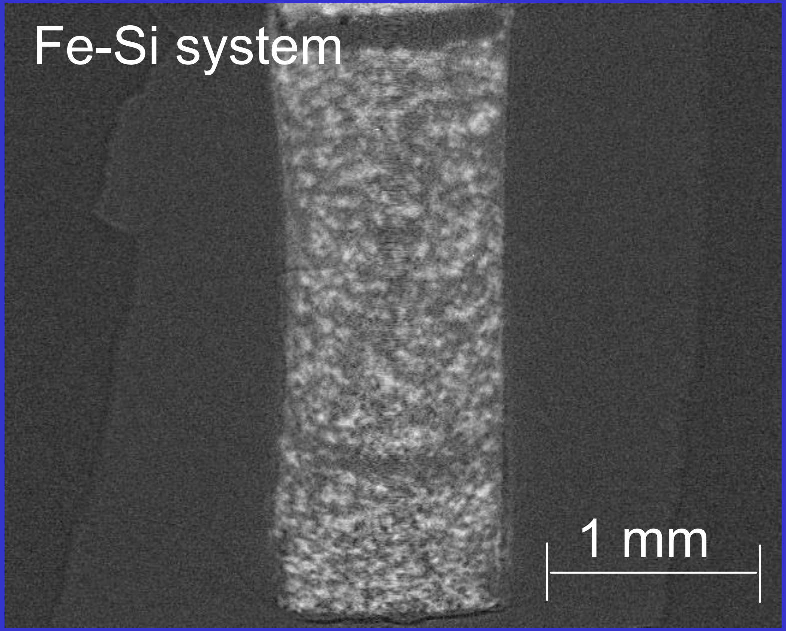


Diffraction and Imaging Setup

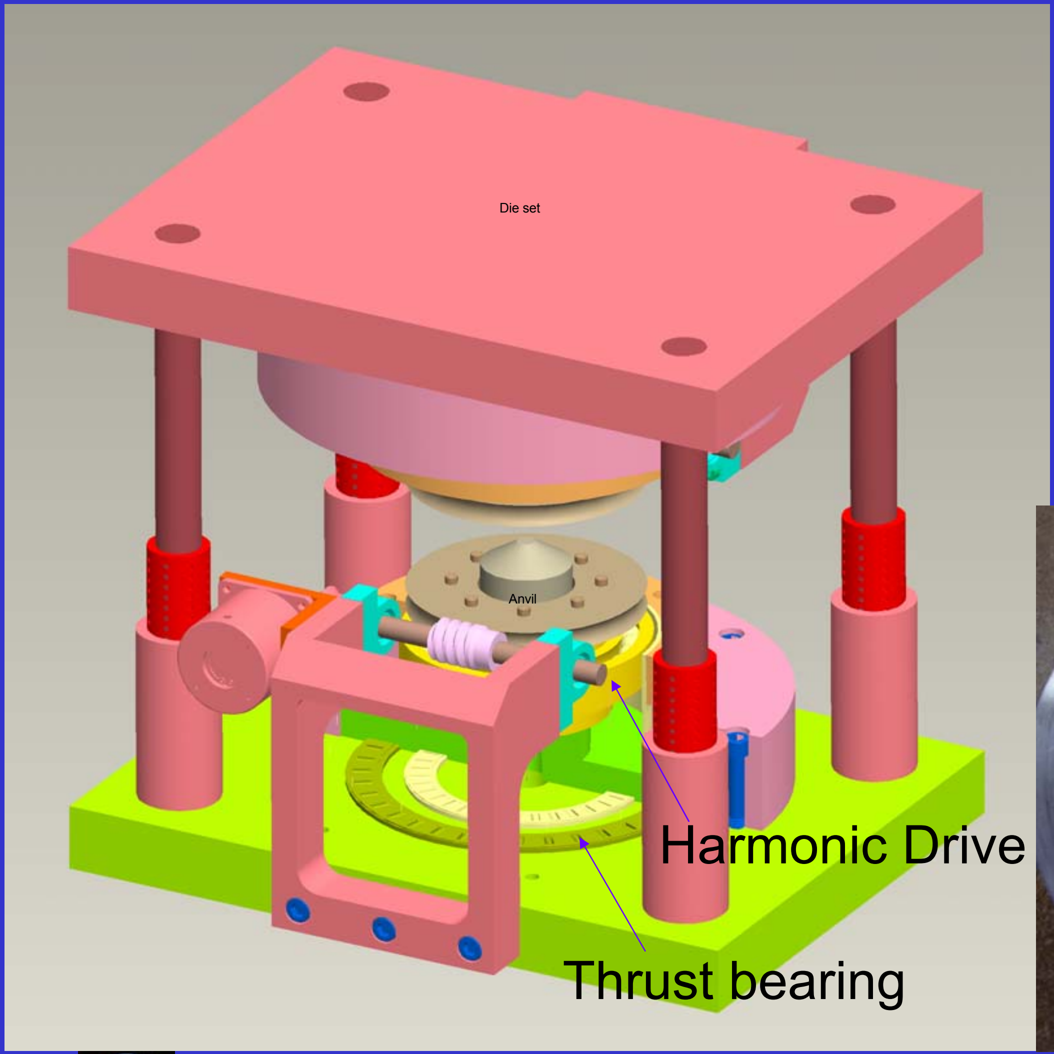


Tomography on Recovered Samples





Rotation Mechanism



Potential Applications

1. High-pressure tomography
   - -In situ observations of Fe-rich melts segregate from silicates
   - -Effects of deformation on melt texture evolution
   - -Density of melts/liquids
2. Volume measurements of liquids/melts
   - -Structure of melts/liquids
   - -Preferred orientation in deformed solids
3. Neutrons